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Indium Nitride Nanostructures Prepared by Various Growth Techniques

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Abstract

In the last few years the interest in the material properties of Indium Nitride (InN) semiconductor has been remarkable. There have been significant improvements in the properties and growth methods of InN nanowires (NWs). High quality single crystalline InN NWs with high growth rate are regularly obtained. InN NWs exhibit a highly conducting quasi two-dimensional electron gas (2DEG) on their surface, which causes nearly metallic conductivity even at low temperatures. The newly verified narrow band gap (~0.69 eV) of InN extends the spectral range covered by III-nitrides to near infrared, which offers a great advantage of nitrides for optoelectronic applications. In this article, the work accomplished in the InN NW research, from its evolution to till now, is reviewed. We mainly concentrate on the growth, characterization, and recent developments in the InN NW research. The most popular growth techniques, metal-organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE) are discussed in detail with their recent progress. Important phenomena in the growth of InN NWs as well as the problems remaining for future study are also discussed.

Keywords: *Metalorganic Deposition, Nanostructures; Optical properties; X-ray diffraction (XRD).*

1. INTRODUCTION

Semiconductor nanowires deserve over growing attention and increasing interest of the scientific community both because of their potential to build future electronic systems and because of intriguing new physical effects which can lead to new functionalities (Taniyasu *et al.* 2006; Ariyawansa *et al.* 2006; Adachi *et al.* 2010; Zhao *et al.* 2011; Babichev *et al.* 2013; Adachi *et al.* 2014; Pust *et al.* 2015). Nanomaterials are the basic building blocks for nanodevices (Li *et al.* 2006; Li *et al.* 2012; Yu *et al.* 2013; Le *et al.* 2014). Therefore, research about nanomaterials is important for both fundamental physics and device application (Li *et al.* 2006; Li *et al.* 2012; Yu *et al.* 2013; Le *et al.* 2014). Due to the distinctive properties of nanostructures, various kinds of InN nanostructures have been synthesized such as nanowires (Yu *et al.* 2015), nanotubes (Wang *et al.* 2015), nanorods (Jiang *et al.* 2009) and nanotips (Schley *et al.* 2007) by solvothermal (Sakalauskas *et al.* 2010), chemical vapor deposition (CVD) (Xu *et al.* 2010; Huang *et al.* 2010; Lei *et al.* 2012), metalorganic chemical vapor deposition (MOCVD) (Chen *et al.* 2009), localized laser-assisted metal organic vapor phase epitaxy (Kim *et al.* 2019), chemical beam epitaxy (CBE) (Chao *et al.* 2006), molecular beam epitaxy (MBE) (Richter *et al.* 2009) and plasma-assisted molecular beam epitaxy (PAMBE) (O'Leary *et al.* 1998). Although many methods have been employed to synthesize one dimensional (1D) InN nanostructures, these methods have their own limitations and drawbacks. InN, however, is not all that easy, even considering the general difficulties encountered in the nitride semiconductor system, to synthesize. The somewhat intractable problem with InN is the enormous difference in the ionic size of its constituent atoms in that the atomic radii for In and N are largely different, which leads to highly distorted interatomic distances, interatomic bonding charges, tendency to form metallic clusters of the group III constituent, and inhomogeneous strain.

2. PROPERTIES OF InN

Indium nitride is a very important III-nitride semiconductor material with many potential applications. The recent publications shows that the blue light emitting diodes (LEDs), photodetectors, laser diodes (LDs), and high electron mobility transistors proved the benefits of nitride material system (Taniyasu *et al.* 2006; Li *et al.* 2006; Ariyawansa *et al.* 2006;

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Adachi *et al.* 2010; Zhao *et al.* 2011; Li *et al.* 2012; Babichev *et al.* 2013; Adachi *et al.* 2014; Le *et al.* 2014; Pust *et al.* 2015). Moreover, due to the large piezoelectricity of III-nitrides, these materials were considered as potential candidates for flexible optoelectronic devices (Yu *et al.* 2013; Wang *et al.* 2015; Yu *et al.* 2015). Additionally III-nitride materials are direct bandgap semiconductors with wide bandgap range (0.68 – 6.2 eV) (Schley *et al.* 2007; Jiang *et al.* 2009; Sakalauskas *et al.* 2010).The InGaN quantum wells are very much essential for light emitting devices because addition of small amount of In in the active GaN layer greatly increases the luminesce efficiency. The ensemble Monte Carlo method reveals that InN exhibits an extremely high drift velocity at room temperature (O'Leary *et al.* 1998; Bellotti *et al.* 1999; Foutz *et al.* 1999). It was found that the transport characteristics of InN are superior to those of GaN and GaAs, over a large range of temperatures from 150 to 500 K.It was identified that InN-based field-effect transistors (FETs) have an extremely high speed with a cutoff frequency of over 1 THzfor 0.1 µm gates. Thus, InN becomes the highly potential material for the fabrication of high-speed high-perform ancehetero junction FETs. The most commonly mentioned band gap value for InN is 1.89 eVat room temperature (Tansley *et al.* 1986). The LEDs, laser diodes (LDs) and transistors commonly involve InGaN, with low in fractions. However, incorporating large fraction of Ininto InGaNleads to great advantages over various applications. The use of InN-based optoelectronic devices extends the potential of an environmental-friendly red emitter with no toxic element, it may replace GaAs-based devices. In addition to that InN is a promising material for low-cost solar cells with high efficiency. In optimum fabrication conditions, the combination of InN and Si with band gap energies 1.9 eV and 1.1 eV leads to obtain a conversion efficiency of over 30 %. The InN/Si solar cell material contains no poisonous materials such as arsenic and it doesn't need phosphine during fabrication process. Qiann et al., reported that InN as a good plasma filter material for the largely used GaSb and GaInAsSb photovoltaic cells in thermophotovoltaic system (Qian *et al.* 2002). Several groups studied the photoluminescence property of single crystalline InN and reported that the band gap energy of InN is lower than 1.0 eV (Matsuoka *et al.* 2002; Wu *et al.* 2002; Johnson *et al.* 2004; Grandal *et al.* 2005). The above newly reported small band gap values are compatible with the wavelength of the optical fiber. Despite the careful scientific effort, one of the basic properties of InN, the band gap is still under debate. Based on the many studies report edit is concluded that the optical properties of InN are very much dependent on the deposition method and the intrinsic doping concentration.

3. DEPOSITION TECHNIQUES OF InN NANOWIRES

Unfortunately, high quality InN is very difficult to synthesize. The growth process involves very low decomposition temperature (~630°C) and high equilibrium vapor pressure of nitrogen.The growth of nanowires generally occurs above the eutectic temperature of the metal catalyst and the semiconducting material. There are many reports on InN films and especially on one dimensional InN nanostructures. In recent years, the number of reports concerning InN research has increases significantly. Vapor-solid growth of InN nanowires using a mixture of In metal/In_2O_3 powder and ammonia was reported by Zhang *et al.* 2002. Other InN nanostructure synthesis methods use a solvo-thermal method, halide chemical vapor deposition, CVD using single source precursors $N_3In[(CH_2)_3NMe_2]_2$, and amonolysis of indium oxide. Among many other growth techniques, metalorganic vapor phase epitaxy (MOVPE), Metalorganic chemical vapor deposition (MOCVD) and molecular beam epitaxy are the most popular growth techniques. The VLS mechanism has proven to be extremely flexible and allows for the controlled growth of complex nanostructures. Nanowires generally grow in the crystal direction that minimizes the total free energy which, in many cases, is dominated by the surface free energy of the interface between the semiconductor and metal catalyst.

Guosheng Cheng *et al.* 2005 fabricated indium nitride nanowires by catalyst free method. They have synthesized InN nanowires at large scale in a quartz tube containing the reagents of indium and indium oxide powders placed in a central part of the hot wall chemical wall deposition (CVD) chamber. Argon gas was initially flown at a constant flow rate of 1 atmosphere base pressure and then the tube was heated upto 700 °C. Upon reaching the temperature of 700°C 100 sccm ammonia was introduced and allowed to flow for 30 minutes continuously. Then the furnace was allowed to cool down naturally at room temperature in an ammonia atmosphere.

Li *et al.* 2016 focused the InN nanowire growth on metal substrates. Due to the vast application of InN nanowires on electronic devices, near-infrared optoelectronics, high-efficiency solar cells and nanogenerators, growing InN nanowires on cheap metal substrate is very attractive. Also this will create a path for various low-cost InN based optoelectronic devices. In this work, the authors designed a homemade metal-organic chemical vapor deposition (MOCVD) system, to grow InN nanowires. They kept the furnace in the atmospheric pressure during the entire growth process and used brass foils as substrates.Trimethylindium and

ammonia were used as precursors and the flow rates were maintained at 14 µmol/min and 3 SLM, respectively. The authors were able to grow high density InN nanowires on brass substrates via MOCVD method.

Chen *et al.* 2012 prepared the InN epitaxial films and nanorods on GaN template by radio-frequency metalorganic molecular beam epitaxy (RF-MOMBE) method.The authors claim that that the growth rate of RF-MOMBE is much higher than the conventional Molecular Beam Epitaxy method and also it is capable of producing high-quality InNnanorods. They prepared the InN films/nanorods on GaN deposited c-plane sapphire substrate by MOMBE system with radio frequency (RF) source to activate the nitrogen. Initially the authors grown 4-µm thick GaN template using commercially available MOCVD system. A turbo molecular pump was used to evacuate the growth chamber with a base pressure of $1x10^{-9}$Torr. During the InNnanorod growth, the N_2flow rate was fixed at 1 sccm (cubic centimeter per minute at STP) and the V/II ratio was adjusted to 0.4 sccm. The thermal cleaning as carried out by heating the substrate for 600 ℃ and then it was cooled down to 500 ℃ for nitradation. The authors used a mass flow controller to control the V/III flow rate.

Lei *et al.* 2012 synthesized InN nanowires in a vertical tubular furnace with straight alumina tube. They used high-purity InN powders and 4H–SiC single crystals as source material and substrates, respectively. In their experiment, 4H–SiC substrates were first eroded by NaOH at 400 °C for 10 min. Then, the eroded substrates and the InN powders were put into the furnace. The remaining air molecules were removed by flushing Ar three times into the furnace. After flushing the Ar into the furnace, the mixed atmosphere of NH3 and N2 was maintained at 1400℃ temperature for 1 hour.Finally the furnace was cooled to the room temperature, and the authors could observe gray color products on the 4H–SiC substrates. The authors observed that the nanowire growth process followed self-catalytic vapor-liquid-solid (VLS) mechanism. In the VLS method, the InN nanoparticles act as a catalyst for 1D growth process.

Song *et al.* 2019 synthesized nanotubes and nanobelts by chemical vapor deposition (CVD) method. The authors reported various InN nanostructures on silicon (100) substrate using CVD method. During the growth process they varied the temperature in a very small range between 700 ℃ to 735 ℃. The morphological studies of the as grown nanotubes and nanobeltsconfirmed the wurtzite crystallographic structure with the preferred orientation along [0001]. Also the room temperature photoluminescence (RT-PL) spectrograph reveals a blue shift. The authors concluded that the temperature is a very sensitive factor thataffects the morphological evolution of the as-prepared InN and other nitride nanostructures.

Stress-free InN nanowires were grown on graphene substrates using sublimation method by Chen *et al.* 2018. The authors used In-metal sublimation CVD method, at normal atmospheric pressure. They adopted this method due to the low cost and effective growth rate. In this process, In-metal was evaporated and allowed to pass through a small gap, after that allowed to deposit on an inverted substrate in NH_3 atmosphere. The authorsfabricated the InN nanowires on graphene/GaN/sapphire, Au/GaN/sapphire and Au/graphene/GaN/sapphire substrates. In this paper, the authors confirmed the growth of InN nanowires was assisted by Au catalyst and also, it is free from stress by introducing graphene inter layer. This graphene layer was prepared on GaN/Sapphire substrate and heated at 120℃ for 15 min to improve the bonding between the substrate and graphene.

Dwivedi *et al.* 2017 demonstrated the synthesis of InNnanopine array using Oblique Angle Deposition (OAD) technique on Si substrate. They have found the average length of the as-synthesized nanopine was ~2μm. The authors proposed a special technique to synthesis well aligned InN nanostructures using customized horizontal quartz tube by OAD method.They mixed high purity Indium Oxide (In_2O_3) powder and Indium (In) powder in a quartz boat in 1:2 wt ratio. One end of the quartz boat was placed in hot furnace and the other end, RCA cleaned p-type Si <100> substrate was placed at an orientation of 85°.After evacuated the quartz tube, they flushed the high purity nitrogen gas into the tube for 30 min. The hot zone was kept at an evaporation temperature of 920-930℃ with a heating rate of 30℃/min.The temperature of the cold zone was kept at 525-530℃ a slow heat increase rate of 15℃/min. The entire growth process was continued for 30 min under the flow of high purity ammonia gas. During the growth process the furnace pressure was maintained at 0.3 mbar. Finally the authors achieved catalyst free high quality InNnanopine structures in a horizontal quartz tube by assembling InNnano pillars of average diameter of ~50 nm.

Kumar *et al.* 2014 reported the growth of InNnanorods by Plasma Assisted Molecular Beam Epitaxy (PAMBE) method. Prior to the initiation ofInN growth an electron beam evaporated Au (~2 nm) layer was deposited in n-Si(111)substrates. The base pressure was maintained below $1 \times 10^{-10}\, mbar$. The Si substrate was chemically cleaned and the deposited Au film was annealed for 600℃ and then the substrate

temperature was decreased to 500℃ to fabricate the nanorods. During the growth process, the nitrogen gas pressure was maintained at $5.8 \times 10^{-5} mbar$.The entire nanorod growth was taken place for 2 hours. The annealed Au film was converted into small dots with an average size range of 15-25 nm. The length of the nanorods grown in this process was in the range of 200-350 nm with average base diameter of ~42 nm.

Barick *et al.* 2015 reported the structural and electronic properties of InN nanowire network synthesized by Chemical Vapor Deposition (CVD) method. The authors prepared the InN nanowires on Au coated quartz substrate using vapor-liquid-solid mechanism. Initially they deposited the gold layers for different thickness (5, 15, and 52 nm) using four target electron beam evaporator. These gold plated substrates were placed side wise on a quartz boat together with high purity indium metal. Mass flow controllers were used to control the Argon and ammonia gas. The flow rates of argon and ammonia gases were maintained at a rate of 20 and 40 sccm respectively. To remove the unwanted gas residues Argon gas was allowed to flow through the quartz tube for about 20 minutes prior to the growth process. Their study reveals the multi nucleation-growth, in that each site is acts as the origin of several nanowires growth results to achieve long and thin InN nanowires. Also the authors conclude that these type of nanowire growth is possible during a specific growth temperature and gold layer thickness. The authors observe that the nanowires prepared in this method grow along $[11\bar{2}0]$ direction (a-plane) to form a dense nanostructure network.

Zhao *et al.* 2013 reported the Mg doped growth of InN nanowires. Before the nanowire growth, a thin In seeding layer is deposited. At higher growth temperatures, the In seed layer turns into In droplets, which can activate the formation of Mg-doped InN nanowires. The growth parameters include a nitrogen flow rate of 1 sccm, a nitrogen plasma power of 350 W, an In flux of 6×10^{-8} Torr, and a substrate temperature of 480 ℃.

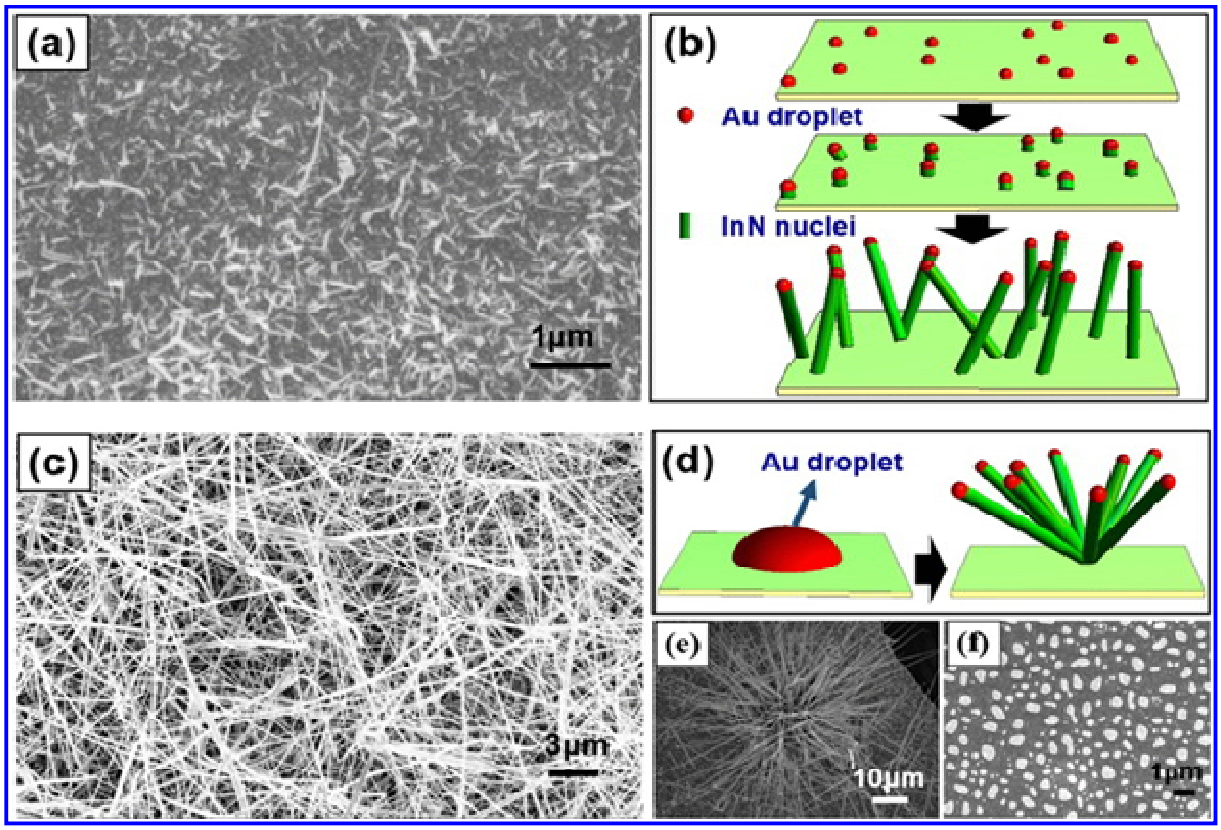

Fig. 1: SEM micrograph of InN (a) grown on 3 nm Au coated quartz substrate at 550 ◦C, (b) schematic diagram of mononuclear growth of nanowires, (c) SEM micrograph of InN grown on 15 nm Au coated quartz substrate at 550 ◦C, (d) schematic diagram of multiple nucleation of nanowires, (e) portion of the SEM micrograph shown in Fig. (c) where multiple nucleation of InN nanowires is clearly visible and (f) SEM image of 15 nm Au coated Si substrate after annealing at 550 ◦C for 30 minutes (Barick *et al.* 2015)

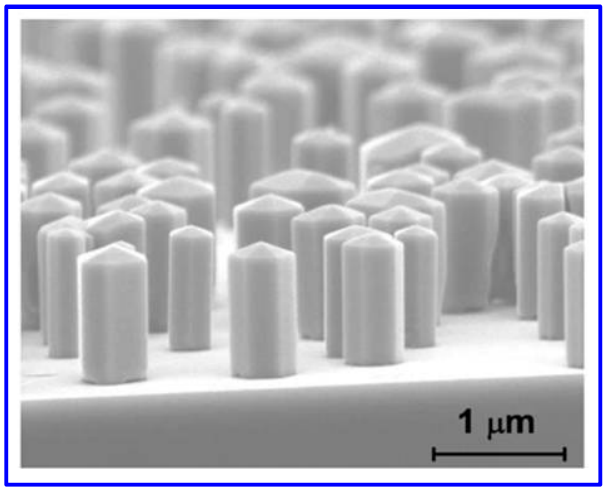

Fig. 2: SEM image of Mg-doped InN nanowires (Zhao *et al.* 2013)

From the fig. 2. we could clearly observe that hexagonal-shaped nanowires are formed. Their characterization further reveals that the InN nanowires synthesized in this method are free of stacking faults and misfit dislocations.

Teker *et al.* 2017 fabricated the InN nanowires by hot-wall 25-mm Low Pressure Chemical Vapor Deposition (LPCVD) method. They have used Si and SiO_2/Si substrates along with Ni catalyst during the growth process. They have ultrasonically cleaned the substrate in acetone, isopropyl alcohol, de-ionized water and finally the substrates were dried with nitrogen. A quartz boat containing both the substrate and In was loaded into CVD reactor and heated to target growth temperature and carrier gas. After that ammonia and hydrogen gasses were applied through the reactor for about 80 min. The gas flow rates were controlled by mass flow controllers and at the end the reactor was cooled down under hydrogen flow until 250 ℃, and then cooled down to room temperature. The authors have observed significant changes in Ni-coated Si substrate and Ni particle applied SiO_2/Si substrate. They have noticed that the high density of nanowires growth was achieved at 1100 ℃. Also they identified the growth rate was high in Ni film catalyst.

Winden *et al.* 2013 reported the site-controlled growth of InN nanostructures using metalorganicvapour phase epitaxy (MOVPE).The authors explained how to obtain selective area growth of InN nanostructures on patterned SiO2/GaN(0001)/c-plane $\alpha - Al_2O_3$ substrates. The authors have used a reactor equipped with in situ optical sensor which is used for temperature determination.GaN layers on *c*-plane sapphire substrates were masked using SiO2. Using standard electron beam lithography and reactive ion etching (RIE) a hexagonally arranged hole array was defines on the sapphire substrate. It is a well understood from the selective area growth the mask geometry influences the surface diffusion as well as vapour phase depletion. The authors used trimethylindium (TMIn) and ammonia (NH3) as the precursors and pure nitrogen (N2) as the carrier and ambient gas. They maintained a chamber pressure of 400 mbar and the growth temperature was varied between 520-720 ℃. The optimized growth conditions were employed to study the nanostructure evolution.The authors concluded that pyramidal shaped nanostructures were obtained. The gradual reduction of growth times gives clear information on how the nanostructures were evolved inside 100 nm mask.

4. CONCLUSION

III-nitride nanostructures have been identified as critical foundations for several nanoscale optoelectronic devices due to their excellent optical and transport properties. InN was the last studied III-nitride material among the alloy family.Among many other nanostructures InN stand out best in fabrication high efficiency solar cells, terahertz emitters and detectors with greater performance due to its large drift velocity at room temperature. Initial growth of InN was performed using powder or small crystals by the method of interacting indium compound with ammonia or thermal decomposition of complex compounds containing In and N.InN nanostructures synthesized by different methods were stressed in this review. From the above review we could clearly observe that the InN NWs fabricated using a catalyst-free molecular beam epitaxy method or a catalyst-assisted CVD process revealed a high crystalline quality. Based the presented results there are a lot of specific growth studies revealed many additional details. The deep knowledge acquired in this field thus allows us to confidently address the challenge of growing very complex heterostructures for future nanodevices.

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